

U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE		ATTORNEY DOCKET NO.
<b>TRANSMITTAL LETTER TO THE UNITED STATES</b>		7393/72546
<b>DESIGNATED/ELECTED OFFICE (DO/EO/US)</b> <b>CONCERNING A FILING UNDER 35 USC 371</b>		U.S. APPLICATION NO.
INTERNATIONAL APPLICATION NO. PCT/EP00/06016		10/030223
		INTERNATIONAL FILING DATE PRIORITY DATE CLAIMED 8 July 1999

## TITLE OF INVENTION

PREPARATION OF SUGAR-FREE HARD CANDY

## APPLICANT(S) FOR DO/EO/US

Michel Henri André GONZE &amp; Freddy Maurits Luc VAN DER SCHUEREN

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1.  This is a **FIRST** submission of items concerning a filing under 35 USC 371.
  2.  This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 USC 371.
  3.  This is an express request to begin national examination procedures (35 USC 371(f)).
  4.  The US has been elected by the expiration of 19 months from the priority date (PCT Article 31).
  5.  A copy of the International Application as filed (35 USC 371(c)(2))
    - a.  is attached hereto (required only if not communicated by the International Bureau).
    - b.  has been communicated by the International Bureau.
    - c.  is not required, as the application was filed in the United States Receiving Office (RO/US).
  6.  An English language translation of the International Application as filed (35 USC 371(c)(2)).
  7.  Amendments to the claims of the International Application under PCT Article 19 (35 USC 371(c)(3))
    - a.  are attached hereto (required only if not communicated by the International Bureau).
    - b.  have been communicated by the International Bureau.
    - c.  have not been made; however, the time limit for making such amendments has NOT expired.
    - d.  have not been made and will not be made.
  8.  An English language translation of the amendments to the claims under PCT Article 19 (35 USC 371(c)(3)).
  9.  An oath or declaration of the inventor(s) (35 USC 371(c)(4)).
  10.  An English language translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 USC 371(c)(5)).
  11. Nucleotide and/or Amino Acid Sequence Submission
    - a.  Computer Readable Form (CRF)
    - b. Specification Sequence Listing on:
      - i.  CD-ROM or CD-R (2 copies); or
      - ii.  Paper Copy
    - c.  Statement verifying identity of above copies
- Items 12 to 19 below concern other document(s) or information included:**
12.  An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
    - a.  Form PTO-1449
    - b.  Copies of Listed Documents
  13.  An assignment for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
  14.  A **FIRST** preliminary amendment.  
 A **SECOND** or **SUBSEQUENT** preliminary amendment.
  15.  A substitute specification.
  16.  A change of power of attorney and/or address letter.
  17.  Application Data Sheet Under 37 CFR 1.76
  18.  Return Receipt Postcard
  19.  Other items or information: PCT Int'l. Search Report; Copies of PCT-Amended Specification pages 3, 4, 4a, 5, 5a, 7, 8; and PCT-Amended Claims

U.S. APPLICATION NO.	INTERNATIONAL APPLICATION NO.	ATTORNEY DOCKET NO.																
10/030223 PCT/EP0/06016		7393/72546																
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<p>20. <input checked="" type="checkbox"/> The following fees are submitted:</p> <p><b>Basic National Fee (37 CFR 1.492(a)(1)-(5)):</b></p> <p>Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO ..... \$1,040.00</p> <p>International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO ..... \$ 890.00</p> <p>International preliminary examination fee (37 CFR 1.482) paid to USPTO, but international search fee (37 CFR 1.445(a)(2)) paid to USPTO ..... \$ 740.00</p> <p>International preliminary examination fee paid to USPTO (37 CFR 1.482) but all claims did not satisfy provisions of PCT Article 33(1)-(4) ..... \$ 710.00</p> <p>International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(1) to (4) ..... \$ 100.00</p>																		
<b>ENTER APPROPRIATE BASIC FEE AMOUNT=</b> \$1,040.00																		
<p>Surcharge of \$130.00 for furnishing the National fee or oath or declaration later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date</p> <table border="1" style="width: 100%;"> <thead> <tr> <th>CLAIMS</th> <th>NUMBER FILED</th> <th>NUMBER EXTRA</th> <th>RATE</th> </tr> </thead> <tbody> <tr> <td>Total Claims</td> <td>10 - 20 =</td> <td>0</td> <td>x \$ 18.00 \$0.00</td> </tr> <tr> <td>Independent Claims</td> <td>2 - 3 =</td> <td>0</td> <td>x \$ 84.00 \$0.00</td> </tr> <tr> <td colspan="2"><input checked="" type="checkbox"/> Multiple Dependent Claim(s) (if applicable)</td> <td></td> <td>+\$280.00 \$280.00</td> </tr> </tbody> </table>			CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE	Total Claims	10 - 20 =	0	x \$ 18.00 \$0.00	Independent Claims	2 - 3 =	0	x \$ 84.00 \$0.00	<input checked="" type="checkbox"/> Multiple Dependent Claim(s) (if applicable)			+\$280.00 \$280.00
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<p><input type="checkbox"/> Applicant claims small entity status. See 37 CFR 1.27. The fees indicated above are reduced by 1/2.</p> <table border="1" style="width: 100%;"> <thead> <tr> <th colspan="2">SUBTOTAL=</th> <th>\$</th> </tr> </thead> <tbody> <tr> <td colspan="2">Processing fee of \$130.00 for furnishing English Translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date.</td> <td>\$</td> </tr> </tbody> </table>			SUBTOTAL=		\$	Processing fee of \$130.00 for furnishing English Translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date.		\$										
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<p>Fee for recording the enclosed assignment. The assignment must be accompanied by an appropriate cover sheet. \$40.00 per property + \$</p> <table border="1" style="width: 100%;"> <thead> <tr> <th colspan="2">TOTAL FEE ENCLOSED=</th> <th>\$1,320.00</th> </tr> </thead> <tbody> <tr> <td colspan="2"></td> <td>Amount to be refunded \$</td> </tr> <tr> <td colspan="2"></td> <td>charged \$</td> </tr> </tbody> </table>			TOTAL FEE ENCLOSED=		\$1,320.00			Amount to be refunded \$			charged \$							
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<p>a. <input type="checkbox"/> A check in the amount of \$ to cover the above fee is enclosed.</p> <p>b. <input checked="" type="checkbox"/> Please charge Deposit Account No. 06-113, Order No. 7393/72546, in the amount of \$1,320.00 to cover the above fees. A duplicate copy of this sheet is enclosed.</p> <p>c. <input checked="" type="checkbox"/> The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 06-1135. A duplicate copy of this sheet is enclosed.</p>																		
<p><b>NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.</b></p>																		
<p>SEND ALL CORRESPONDENCE TO:</p> <p>Kendrew H. Colton, Reg. No. 30,368      Fitch, Even, Tabin &amp; Flannery      Suite 1600, South LaSalle Street      Chicago, Illinois 60603-3406      (202) 419-7000 (telephone)      (202) 419-7007 (facsimile)</p> <p>      Kendrew H. Colton, Registration No. 30,368      One of the Attorneys for Applicant(s)</p> <p>January 8, 2002      Date</p>																		

**PATENT**

Attorney Docket No. 7393/72546

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

In re Application of:

GONZE et al.

Application No.: (Not Yet Assigned)

Filed: Herewith

For: PREPARATION OF SUGAR-FREE HARD CANDY

January 8, 2002

**PRELIMINARY AMENDMENT AND COPY OF AMENDED  
PAGES/CLAIMS OF PCT INT'L. APPLICATION**

Commissioner for Patents  
Washington, D.C. 20231

Dear Sir:

Please amend this application ad follows:

**IN THE SPECIFICATION:**

At the top of the first page, just under the title, insert

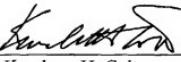
This application is the National phase of International Application PCT/EP00/06016, filed 28 June 2000, which designated the U.S. and that International Application was published under PCT Article 21(2) in English, the disclosure of which is incorporated herein by reference.

**REMARKS**

Please enter this Preliminary Amendment plus the attached amended PCT specification pages and the PCT amended claims.

Respectfully submitted,

FITCH, EVEN, TABIN & FLANNERY

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Registration No. 30,368

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used for the sugar-less foodstuffs, one or more sugar alcohols selected from the group consisting of lactitol, palatinol and maltitol may preferably be used as a sweetener.

A process for producing hard candies comprises preparation of syrups, cooking at elevated temperature and shaping including a cooling phase. The mass viscosity of the hot (temperature > 90°C) syrups is determining the workability and the processing time of the cooling phase. Applying low viscous syrups results during the cooling phase of the shaping of hard candies in a very liquid mass, which needs a considerably long processing time before the mass can be shaped in moulds.

Accordingly, there is a need for highly viscous syrups which are non-hygroscopic and which can be used for the preparation of i.e. sugar-free non-hygroscopic, non-sticky and non-cariogenic hard candies. Applying high viscous syrups shortens the processing time of the cooling phase considerably and it results in an economical advantageous process.

Summary of the invention

The present invention discloses a syrup (1) having a dry substance of from 60% to 80% consisting of a mixture of hydrogenated starch hydrolysate syrup (2) and isomalt powder or isomalt syrup (3) characterised in that:

- a) the dry substance of the syrup (1) consists of from 14 to 25% w/w hydrogenated starch hydrolysate syrup (2) (dry substance) wherein the dry substance of said hydrogenated starch hydrolysate syrup (2) comprises between 22% w/w and 55% w/w higher polyols, and
- b) the dry substance of the syrup (1) consists of from 75 to 86% w/w isomalt, and
- c) the dry substance of the syrup (1) consists of:  
7 % w/w to 52% w/w 6-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,6 GPS),  
24.5% w/w to 52% w/w 1-O- $\alpha$ -D-glucopyranosyl-D-mannitol (1,1 GPM),  
0% w/w to 52% w/w 1-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,1 GPS),  
0% w/w to 1.3% w/w sorbitol (DP<sub>1</sub>),  
2.8% w/w to 13.8% w/w maltitol (DP<sub>2</sub>),  
1.5% w/w to 4.2% w/w maltotriitol (DP<sub>3</sub>),  
3.0% w/w to 13.5% w/w higher polyols (DP<sub>n</sub>)

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The present invention further relates to a syrup having aforementioned composition and having a dry substance of 70%.

These syrups are used for the preparation of sugar-free hard candies, which are transparent, non-hygroscopic, non-cariogenic and non-sticky.

The present invention further relates to a sugar-free non-cariogenic hard candy with a mass viscosity of at least 25,000 mPa.s, and a candy surface stickiness below 180 g (50% RH, 22-23°C) and a moisture pick-up (hygroscopicity at 70% R.H and 25°C) after 14 days below 1.5%.

The current invention relates to a process for the production of a sugar-free non-cariogenic hard candy comprising the following steps:

- a) preparing a syrup (1) having a dry substance of from 60% to 80% consisting of a mixture of hydrogenated starch hydrolysate syrup (2) and isomalt powder or isomalt syrup (3) characterised in that the dry substance of the syrup (1) consists of from 14 to 25% w/w hydrogenated starch hydrolysate syrup (2) (dry substance) wherein the dry substance of said hydrogenated starch hydrolysate syrup (2) comprises between 22% w/w and 55% w/w higher polyols, and the dry substance of the syrup (1) consists of from 75 to 86% w/w isomalt, and the dry substance of the syrup (1) consists of 7 % w/w to 52% w/w 6-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,6 GPS), 24.5% w/w to 52% w/w 1-O- $\alpha$ -D-glucopyranosyl-D-mannitol (1,1 GPM), 0% w/w to 52% w/w 1-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,1 GPS), 0% w/w to 1.3% w/w sorbitol (DP<sub>1</sub>), 2.8% w/w to 13.8% w/w maltitol (DP<sub>2</sub>), 1.5% w/w to 4.2% w/w maltotriitol (DP<sub>3</sub>), 3.0% w/w to 13.5% w/w higher polyols (DP<sub>n</sub>),
- b) cooking at a temperature between 145°C and 170°C and low vacuum, and
- c) shaping according to stamping or deposit method.

The current invention further relates to a process wherein flavour and/or colouring matter is added during shaping.

The current invention discloses a process wherein the syrup comprises between 10 to 35% by weight water.

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The current invention further relates to a process wherein the water content of the hard candy is less than 4% by weight, preferably less than 2% by weight or less, based upon the weight of the hard candy.

Non-sticky, non-hygroscopic and non-cariogenic sugar-free hard candies are obtained.

Brief description of the drawings

Figure 1 shows the mass viscosity profile of the hard candies prepared with C $\star$  Maltidex C16510, which is a syrup at 70% dry substance and consisting of 70/30 isomalt/hydrogenated starch hydrolysate mixture (= 70% hydrogenated isomaltulose

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powder (C★ Maltidex C16500) and 30% hydrogenated starch hydrolysate syrup added at commercial base (=70% dry substance) and wherein the dry substance of hydrogenated starch hydrolysate is comprising between 22% and 31% w/w higher polyols)-⊗-, which is compared to the mass viscosity of hard candies prepared from hydrogenated isomaltulose (C★ Maltidex C16500,-), respectively.

Figure 2 shows the hygroscopicity (at 70% relative humidity, and 25°C) of hard candies prepared with hydrogenated isomaltulose (C★ Maltidex C16500, -□-), hydrogenated starch hydrolysate syrup (C★ Maltidex M16311, -○-), and (C★ Maltidex C16510, which is a syrup at 70% dry substance and consisting of 70/30 isomalt/hydrogenated starch hydrolysate mixture (= 70% hydrogenated isomaltulose powder (C★ Maltidex C16500) and 30% hydrogenated starch hydrolysate syrup added at commercial base (=70% dry substance) and wherein the dry substance of hydrogenated starch hydrolysate is comprising between 22% and 31% w/w higher polyols) -⊗-, respectively.

Figure 3 shows the telemetric evaluation of hard candies prepared with C★ Maltidex C16510 (which is a syrup at 70% dry substance and consisting of 70/30 isomalt/hydrogenated starch hydrolysate mixture (= 70% hydrogenated isomaltulose powder (C★ Maltidex C16500) and 30% hydrogenated starch hydrolysate syrup added at commercial base (=70% dry substance) and wherein the dry substance of hydrogenated starch hydrolysate is comprising between 22% and 31% w/w higher polyols) followed by sucrose intake.

Detailed description of the invention

The present invention discloses a syrup (1) having a dry substance of from 60% to 80% consisting of a mixture of hydrogenated starch hydrolysate syrup (2) and isomalt powder or isomalt syrup (3) characterised in that:

- a) the dry substance of the syrup (1) consists of from 14 to 25% w/w hydrogenated starch hydrolysate syrup (2) (dry substance) wherein the dry substance of said hydrogenated starch hydrolysate syrup (2) comprises between 22% w/w and 55% w/w higher polyols, and
- b) the dry substance of the syrup (1) consists of from 75 to 86% w/w isomalt, and
- c) the dry substance of the syrup (1) consists of:

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7 % w/w to 52% w/w 6-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,6 GPS),  
24.5% w/w to 52% w/w 1-O- $\alpha$ -D-glucopyranosyl-D-mannitol (1,1 GPM),  
0% w/w to 52% w/w 1-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,1 GPS),  
0% w/w to 1.3% w/w sorbitol (DP<sub>1</sub>),  
2.8% w/w to 13.8% w/w maltitol (DP<sub>2</sub>),  
1.5% w/w to 4.2% w/w maltotriitol (DP<sub>3</sub>),  
3.0% w/w to 13.5% w/w higher polyols (DP<sub>n</sub>)

The syrups can be obtained by hydrogenating mixtures of starch hydrolysates and isomaltulose or 'isomerised' saccharose, or by mixing the hydrogenated products, which

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15.6 – 16.6% w/w (maltotriitol) and 26.4-28.1% w/w higher polyols (hydrogenated oligomers), and the dry substance is 70%. Whenever the dry substance of the syrup contains at least 50% w/w DP<sub>2</sub>, the syrup is defined as a maltitol syrup.

Isomalt powder is hydrogenated isomaltulose powder and can be obtained by the hydrogenation of isomaltulose which results in a quasi-equimolar mixture of 6-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,6 GPS), and 1-O- $\alpha$ -D-glucopyranosyl-D-mannitol (1,1 GPM). A more typical composition consists of 52.6 – 53.6% w/w 6-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,6 GPS), and 44.7 – 46.6% w/w 1-O- $\alpha$ -D-glucopyranosyl-D-mannitol (1,1 GPM).

Isomalt syrup is hydrogenated ‘isomerised saccharose’ which can be prepared according to a method as is described in EP 0 625 578, and the dry substance of the syrup comprises a mixture of 10% w/w to 50% w/w 6-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,6 GPS), 35% w/w to 60% w/w 1-O- $\alpha$ -D-glucopyranosyl-D-mannitol (1,1 GPM) and 2% w/w to 60% w/w 1-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,1 GPS).

The dry substance of a more typical composition, obtained after the hydrogenation of ‘isomerised’ saccharose, comprises 43.6% w/w 6-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (4,6 GPS), 49.2% w/w 1-O- $\alpha$ -D-glucopyranosyl-D-mannitol (1,1 GPM) and 3.1% w/w 1-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,1 GPS).

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A typical composition of the current invention, i.e. C $\star$  Maltidex C16510 refers to 70/30 isomalt/hydrogenated starch hydrolysate mixture and is which is a syrup at 70% dry substance and consisting of 70% hydrogenated isomaltulose powder (C $\star$  Maltidex C16500) and 30% hydrogenated starch hydrolysate syrup added at commercial base (=70% dry substance) and wherein the dry substance of hydrogenated starch hydrolysate is comprising between 22% and 31% w/w higher polyols) The dry substance of C $\star$  Maltidex C16510 has the following composition:

DP <sub>1</sub> :	0.7 – 1.0 % w/w
DP <sub>2</sub> :	10 - 12% w/w
DP <sub>3</sub> :	2.4 – 4.4% w/w
DP <sub>n</sub> :	4.6 – 6.6 % w/w
1,6 GPS:	41.5 – 42.3 % w/w
1,1 GPM:	35.3 – 36.8 % w/w

These syrups are used for the preparation of sugar-free hard candies, which are transparent, non-hygroscopic, non-cariogenic and non-sticky.

The current invention relates to a process for the production of a sugar-free non-cariogenic hard candy comprising the following steps:

- a) preparing a syrup (1) having a dry substance from 60% to 80% consisting of a mixture of hydrogenated starch hydrolysate syrup (2) and isomalt powder or isomalt syrup (3) characterised in that the dry substance of the syrup (1) consists of from 14 to 25% w/w hydrogenated starch hydrolysate syrup (2) (dry substance) wherein the dry substance of said hydrogenated starch hydrolysate syrup (2) comprises between 22% w/w and 55% w/w higher polyols, and the dry substance of the syrup (1) consists of from 75 to 86% w/w isomalt, and the dry substance of the syrup (1) consists of 7 % w/w to 52% w/w 6-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,6 GPS), 24.5% w/w to 52% w/w 1-O- $\alpha$ -D-glucopyranosyl-D-mannitol (1,1 GPM), 0% w/w to 52% w/w 1-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,1 GPS), 0% w/w to 1.3% w/w sorbitol (DP<sub>1</sub>), 2.8% w/w to 13.8% w/w maltitol (DP<sub>2</sub>), 1.5% w/w to 4.2% w/w maltotriitol (DP<sub>3</sub>), 3.0% w/w to 13.5% w/w higher polyols (DP<sub>n</sub>),

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- b) cooking at a temperature between 145°C and 170°C and low vacuum, and
- c) shaping according to stamping or deposit method.

Production of the hard candies may be carried out in batch evaporators or in continuous cookers and the process time at elevated temperature generally being up to 15 minutes, preferably in the range 5 to 10 minutes.

## **CLAIMS**

1. A syrup (1) having a dry substance of from 60 to 80% consisting of a mixture of hydrogenated starch hydrolysate syrup (2) and isomalt powder or isomalt syrup (3) characterised in that:
    - a) the dry substance of the syrup (1) consists of from 14 to 25% w/w hydrogenated starch hydrolysate syrup (2) (dry substance) wherein the dry substance of said hydrogenated starch hydrolysate syrup (2) comprises between 22 and 55% w/w higher polyols, and
    - b) the dry substance of the syrup (1) consists of from 75 to 86% w/w isomalt, and
    - c) the dry substance of the syrup (1) consists of:
      - 7 to 52% w/w 6-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,6 GPS)
      - 24.5 to 52% w/w 1-O- $\alpha$ -D-glucopyranosyl-D-mannitol (1,1 GPM)
      - 0 to 52% w/w 1-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,1 GPS)
      - 0 to 1.3% w/w sorbitol (DP<sub>1</sub>)
      - 2.8 to 13.8% w/w maltitol (DP<sub>2</sub>)
      - 1.5 to 4.2% w/w maltotriitol (DP<sub>3</sub>)
      - 3.0 to 13.5% w/w higher polyols (DP<sub>n</sub>)
  2. A syrup according to claim 1 characterised in that said syrup has a dry substance of 70%.
  3. A sugar-free non-cariogenic hard candy comprising a syrup according to claims 1 or 2.
  4. A sugar-free non-cariogenic hard candy according to claim 3 characterised in that the mass viscosity is at least 25,000 mPa.s, and that the candy surface stickiness is below 180g (50% RH, 22-23°C) and the moisture pick-up (hygroscopicity at 70% RH and 25°C) after 14 days is below 1.5%.

5. A process for the production of a sugar-free non-cariogenic hard candy comprising the following steps:
  - a) preparing a syrup (1) having a dry substance of from 60 to 80% consisting of a mixture of hydrogenated starch hydrolysate syrup (2) and isomalt powder or isomalt syrup (3) characterised in that the dry substance of the syrup (1) consists of from 14 to 25% w/w hydrogenated starch hydrolysate syrup (2) (dry substance) wherein the dry substance of said hydrogenated starch hydrolysate syrup (2) comprises between 22 and 55% w/w higher polyols, and the dry substance of the syrup (1) consists of from 75 to 86% w/w isomalt, and the dry substance of the syrup (1) consists of 7 to 52% w/w 6-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,6 GPS), 24.5 to 52% w/w 1-O- $\alpha$ -D-glucopyranosyl-D-mannitol (1,1 GPM), 0 to 52% w/w 1-O- $\alpha$ -glucopyranosyl-D-sorbitol (1,1 GPS), 0 to 1.3% w/w sorbitol (DP<sub>1</sub>), 2.8 to 13.8% w/w maltitol (DP<sub>2</sub>), 1.5 to 4.2% w/w maltotriitol (DP<sub>3</sub>), 3.0 to 13.5% w/w higher polyols (DP<sub>n</sub>),
  - b) cooking at a temperature between 145 and 170°C and low vacuum, and
  - c) shaping according to stamping or deposit method.
6. A process according to claim 5 characterised in that flavour and/or colouring matter is added during shaping.
7. A process according to claims 5 or 6 characterised in that the syrup comprises between 10 to 35% by weight water.
8. A process according to any one of claims 5 to 7 characterised in that the water content of the hard candy is less than 4% by weight, preferably less than 2% by weight or less, based upon the weight of the hard candy.

AMENDED SHEET

3/PB

### Preparation of Sugar-free Hard candy

#### Technical field

The present invention relates to mixtures consisting of hydrogenated starch hydrolysate syrup and isomalt powder or isomalt syrup. The dry substance of the hydrogenated starch hydrolysate syrup comprises between 22% w/w and 55% w/w higher polyols. The present invention also discloses a process for producing sugar-free hard candies. In addition, a new type of sugar-free hard candies is disclosed. The candies are non-hygroscopic, non-sticky, non-cariogenic and show excellent mass viscosity.

#### Background of the invention

Recent developments in hard candy manufacture have been the replacement of part or all of the sugar by a sugar alcohol (polyol) in the interest of providing a product having a reduced calorie content and a lower tendency to cause tooth decay. Among the polyols, which have been proposed for the manufacture of hard candy, are isomalt, maltitol, xylitol, erythritol and others.

EP 0 533 334 describes a process for the production of hard candy characterised in that the maltitol content of the sugar alcohol mixture is more than 77% but less than 86% by weight based on dry substance. When the mixture contains 77% or less maltitol the resultant candies become increasingly hygroscopic and sticky, while when the mixture contains 86% and higher maltitol the candies rapidly become opaque as a result of the maltitol crystallisation.

EP 0 611 527 describes a controlled propagation of crystallisation of maltitol in hard candies by applying molecules of molecular weight greater than 1,300 Dalton, preferably greater than 2,000 Dalton and more preferably greater than 3,000 Dalton. Preferably syrups comprising more than 78% maltitol are applied, since syrups comprising less than 60% maltitol give unsatisfactory results in respect to hygroscopicity, cariogenicity and unsuitable application.

US 4,971,798 describes a hydrogenated isomaltulose based hard confection which contains, in addition to the hydrogenated isomaltulose, a medicinal active ingredient. Such a formulation has been found to dissolve more slowly than similar formulations

based on sugar, rendering them suitable for dispensing the active ingredient over an extended period time.

Furthermore, it is common general knowledge that hard candies based on hydrogenated isomaltulose are non-hygroscopic but the mass viscosity of hard candies based on hydrogenated isomaltulose is low.

WO 97/30598 describes a sweetener consisting of especially 6-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,6 GPS), 1-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,1 GPS) and 1-O- $\alpha$ -D-glucopyranosyl-D-mannitol (1,1 GPM), and the use of this sweetener in hard and soft caramels, especially pharmaceutically active caramels. The presence of 1-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,1 GPS) results in candies with improved solubility, higher sweetness and lower ability to crystallise, but hard candies based on these hydrogenated isomerised saccharose syrups have even lower mass viscosity than the hard candies based on hydrogenated isomaltulose.

Fritzsching, in The Manufacturing Confectioner Vol 75, No. 11, 1995, pages 65 – 73, describes isomalt in hard candy applications. He demonstrates that the hard candies prepared with 80% isomalt and 20% of a hygroscopic polyol have a higher hygroscopicity than hard candies prepared with pure isomalt. If isomalt is blended with hygroscopic polyols or bulking agents a more excessive packaging like twist wrapping is required. Due to their slight stickiness, loose packing in boxes is not possible.

DE 4228278 discloses a fondant and its production process. The sugar-free fondant comprises 75 to 90% w/w disaccharide alcohol and 10 to 25% w/w of water. The fondant is used in particular as icing agent, and for fillings of pralines. The disaccharide alcohol can be isomalt, maltitol and the like.

EP 0500977 relates to body and mouthfeel potentiated foods and beverages containing neohesperidin dihydrochalcone and a process for preparing them. It describes the flavour and sweetness profile when applying neohesperidin dihydrochalcone.

EP 0788744 relates to de-lactose milk and de-lactose milk powder which are reconstructed with milk proteins and milk fats as major components free or almost free of lactose. Furthermore, the invention relates to foodstuffs containing de-lactose milk and/or de-lactose milk powder. When the de-lactose milk and the de-lactose milk powder are

used for the sugar-less foodstuffs, one or more sugar alcohols selected from the group consisting of lactitol, palatinose and maltitol may preferably be used as a sweetener.

A process for producing hard candies comprises preparation of syrups, cooking at elevated temperature and shaping including a cooling phase. The mass viscosity of the hot (temperature > 90°C) syrups is determining the workability and the processing time of the cooling phase. Applying low viscous syrups results during the cooling phase of the shaping of hard candies in a very liquid mass, which needs a considerably long processing time before the mass can be shaped in moulds.

Accordingly, there is a need for highly viscous syrups which are non-hygroscopic and which can be used for the preparation of i.e. sugar-free non-hygroscopic, non-sticky and non-cariogenic hard candies. Applying high viscous syrups shortens the processing time of the cooling phase considerably and it results in an economical advantageous process.

#### Summary of the invention

The present invention discloses a syrup at dry substance between 60% to 80%, preferably at dry substance of about 70% consisting of a mixture of hydrogenated starch hydrolysate syrup and isomalt powder or isomalt syrup characterised in that the dry substance of the syrup comprises 86% w/w isomalt, preferably between 85% w/w and 79% w/w isomalt, more preferably 75% w/w isomalt, and the dry substance of the hydrogenated starch hydrolysate syrup comprises between 22% w/w and 55% w/w higher polyols, preferably between 25% w/w and 50% w/w higher polyols.

The present invention further discloses a syrup wherein the dry base of the syrup consists of between:

- 7 % w/w to 52% w/w 6-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,6 GPS),
- 24.5% w/w to 52% w/w 1-O- $\alpha$ -D-glucopyranosyl-D-mannitol (1,1 GPM),
- 0% w/w to 52% w/w 1-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,1 GPS),
- 0% w/w to 1.3% w/w sorbitol (DP1),
- 2.8% w/w to 13.8% w/w maltitol (DP2),
- 1.5% w/w to 4.2% w/w maltotriitol (DP3),
- 3.0% w/w to 13.5% w/w higher polyols (DPn).

These syrups are used for the preparation of sugar-free hard candies, which are transparent, non-hygroscopic, non-cariogenic and non-sticky.

The present invention further relates to a sugar-free non-cariogenic hard candy with a mass viscosity of at least 25,000 mPa.s, and a candy surface stickiness below 180 g (50% RH, 22-23°C) and a moisture pick-up (hygroscopicity at 70% R.H and 25°C) after 14 days below 1.5%.

The current invention relates to a process for the production of a sugar-free non-cariogenic hard candy comprising the following steps:

- a) preparing a syrup at dry substance between 60% to 80% consisting of a mixture of hydrogenated starch hydrolysate syrup and isomalt powder or isomalt syrup characterised in that the dry substance of the syrup comprises 86% w/w isomalt, preferably between 85% w/w and 79% w/w isomalt, more preferably 75% w/w isomalt, and the dry substance of the hydrogenated starch hydrolysate syrup comprises between 22% w/w and 55% w/w higher polyols, preferably between 25% w/w and 50% w/w higher polyols,
- b) cooking at a temperature between 145°C and 170°C and low vacuum, and
- c) shaping according to stamping or deposit method.

The current invention further relates to a process wherein flavour and/or colouring matter is added during shaping.

The current invention discloses a process wherein the syrup comprises between 10 to 35% by weight water.

The current invention further relates to a process wherein the water content of the hard candy is less than 4% by weight, preferably less than 2% by weight or less, based upon the weight of the hard candy.

Non-sticky, non-hygroscopic and non-cariogenic sugar-free hard candies are obtained.

#### Brief description of the drawings

Figure 1 shows the mass viscosity profile of the hard candies prepared with C $\star$  Maltidex C16510, which is a syrup at 70% dry substance and consisting of 70/30 isomalt/hydrogenated starch hydrolysate mixture (= 70% hydrogenated isomaltulose

powder (C $\star$  Maltidex C16500) and 30% hydrogenated starch hydrolysate syrup added at commercial base (=70% dry substance) and wherein the dry substance of hydrogenated starch hydrolysate is comprising between 22% and 31% w/w higher polyols)- $\otimes$ -, which is compared to the mass viscosity of hard candies prepared from hydrogenated isomaltulose (C $\star$  Maltidex C16500,-), respectively.

Figure 2 shows the hygroscopicity (at 70% relative humidity, and 25°C) of hard candies prepared with hydrogenated isomaltulose (C $\star$  Maltidex C16500, -□-), hydrogenated starch hydrolysate syrup (C $\star$  Maltidex M16311, -○-), and (C $\star$  Maltidex C16510, which is a syrup at 70% dry substance and consisting of 70/30 isomalt/hydrogenated starch hydrolysate mixture (= 70% hydrogenated isomaltulose powder (C $\star$  Maltidex C16500) and 30% hydrogenated starch hydrolysate syrup added at commercial base (=70% dry substance) and wherein the dry substance of hydrogenated starch hydrolysate is comprising between 22% and 31% w/w higher polyols) -⊗-), respectively.

Figure 3 shows the telemetric evaluation of hard candies prepared with C $\star$  Maltidex C16510 (which is a syrup at 70% dry substance and consisting of 70/30 isomalt/hydrogenated starch hydrolysate mixture (= 70% hydrogenated isomaltulose powder (C $\star$  Maltidex C16500) and 30% hydrogenated starch hydrolysate syrup added at commercial base (=70% dry substance) and wherein the dry substance of hydrogenated starch hydrolysate is comprising between 22% and 31% w/w higher polyols) followed by sucrose intake.

#### Detailed description of the invention

The present invention discloses a syrup at dry substance between 60% to 80%, preferably at dry substance of about 70% consisting of a mixture of hydrogenated starch hydrolysate syrup and isomalt powder or isomalt syrup characterised in that the dry substance of the syrup comprises 86% w/w isomalt, preferably between 85% w/w and 79% w/w, more preferably 75% w/w isomalt, and the dry substance of the hydrogenated starch hydrolysate syrup comprises between 22% w/w and 55% w/w higher polyols, preferably between 25% w/w and 50% w/w higher polyols.

The syrups can be obtained by hydrogenating mixtures of starch hydrolysates and isomaltulose or 'isomerised' saccharose, or by mixing the hydrogenated products, which

are obtained by hydrogenation of starch hydrolysate and isomaltulose and 'isomerised' saccharose, respectively, until the aforementioned composition is obtained.

Starch hydrolysates are obtained by the enzymatic or acidic hydrolysis of starch and the starch hydrolysate comprises glucose, maltose, maltotriose, and higher glucose oligomers ((DPn where n = >3). A suitable starch hydrolysate comprises between 22% w/w and 55% w/w higher oligomers, preferably between 25% w/w and 50% w/w higher oligomers.

The dry substance of a typical suitable starch hydrolysate syrup consists of:

glucose:	1 – 5% w/w
maltose:	20 – 55% w/w
maltotriose:	11–16.6% w/w
higher oligomers:	22 – 54% w/w

Isomaltulose or 6-O- $\alpha$ -D-glucopyranosyl-D-fructofuranose is synthesised from sucrose by the action of an enzyme present in bacterial strains like *Protaminobacter rubrum*, *Erwinia rhamontici* and *Serratia plymuthica*.

'Isomerised' saccharose is obtained by enzymatic conversion of saccharose (i.e. sucrose) into a saccharide mixture, which is containing trehalulose and isomaltulose.

Hydrogenation of the mixtures of starch hydrolysate and isomaltulose or 'isomerised' saccharose results in mixtures comprising sorbitol, maltitol, maltotriitol, and hydrogenated oligomers (higher polyols), 6-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,6 GPS), and 1-O- $\alpha$ -D-glucopyranosyl-D-mannitol (1,1 GPM), and 1-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,1 GPS).

The components such as sorbitol, maltitol, maltotriitol, and hydrogenated oligomers (higher polyols) are obtained by the hydrogenation of starch hydrolysate syrups. The other components such as 6-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,6 GPS), and 1-O- $\alpha$ -D-glucopyranosyl-D-mannitol (1,1 GPM), and 1-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,1 GPS) are obtained by hydrogenation of isomaltulose and 'isomerised' saccharose.

Hydrogenation of a starch hydrolysate syrup can give a typical composition wherein the dry substance consists of 52.3 – 53.6% w/w maltitol, 3.3-3.9% w/w sorbitol,

Isomalt powder is hydrogenated isomaltulose powder and can be obtained by the hydrogenation of isomaltulose which results in a quasi-equimolar mixture of 6-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,6 GPS), and 1-O- $\alpha$ -D-glucopyranosyl-D-mannitol (1,1 GPM). A more typical composition consists of 52.6 - 53.6% w/w 6-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,6 GPS), and 44.7 - 46.6% w/w 1-O- $\alpha$ -D-glucopyranosyl-D-mannitol (1,1 GPM).

Isomalt syrup is hydrogenated 'isomerised saccharose' which can be prepared according to a method as is described in EP 0 625 578, and the dry substance of the syrup comprises a mixture of 10% w/w to 50% w/w 6-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,6 GPS), 35% w/w to 60% w/w 1-O- $\alpha$ -D-glucopyranosyl-D-mannitol (1,1 GPM) and 2% w/w to 60% w/w 1-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,1 GPS).

The dry substance of a more typical composition, obtained after the hydrogenation of 'isomerised' saccharose, comprises 43.6% w/w 6-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,6 GPS), 49.2% w/w 1-O- $\alpha$ -D-glucopyranosyl-D-mannitol (1,1 GPM) and 3.1% w/w 1-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,1 GPS).

The present invention discloses a syrup and the dry substance thereof consisting of between 14% - 25% w/w hydrogenated starch hydrolysate syrup (dry substance) and between 75% - 86% w/w isomalt and it has the following composition:

- 7 % w/w to 52% w/w 6-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,6 GPS),
- 24.5% w/w to 52% w/w 1-O- $\alpha$ -D-glucopyranosyl-D-mannitol (1,1 GPM),
- 0% w/w to 52% w/w 1-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,1 GPS),
- 0% w/w to 1.3% w/w sorbitol (DP1),
- 2.8% w/w to 13.8% w/w maltitol (DP2),
- 1.5% w/w to 4.2% w/w maltotriitol (DP3),
- 3.0% w/w to 13.5% w/w higher polyols (DPn).

A typical composition of the current invention, i.e. C<sub>16</sub> Maltidex C16510 refers to 70/30 isomalt/hydrogenated starch hydrolysate mixture and is which is a syrup at 70% dry substance and consisting of 70% hydrogenated isomaltulose powder (C<sub>16</sub> Maltidex C16500) and 30% hydrogenated starch hydrolysate syrup added at commercial base (=70% dry substance) and wherein the dry substance of hydrogenated starch hydrolysate is comprising between 22% and 31% w/w higher polyols) The dry substance of C<sub>16</sub> Maltidex C16510 has the following composition:

DP <sub>1</sub> :	0.7 – 1.0 % w/w
DP <sub>2</sub> :	10 - 12% w/w
DP <sub>3</sub> :	2.4 – 4.4% w/w
DP <sub>n</sub> :	4.6 – 6.6 % w/w
1,6 GPS:	41.5 – 42.3 % w/w
1,1 GPM:	35.3 – 36.8 % w/w

These syrups are used for the preparation of sugar-free hard candies, which are transparent, non-hygroscopic, non-cariogenic and non-sticky.

The current invention relates to a process for the production of a sugar-free non-cariogenic hard candy comprising the following steps:

- a) preparing a syrup at dry substance between 60% to 80% consisting of a mixture of hydrogenated starch hydrolysate syrup and isomalt powder or isomalt syrup characterised in that the dry substance of the syrup comprises 86% w/w isomalt, preferably between 85% w/w and 79% w/w isomalt, more preferably 75% w/w isomalt, and the dry substance of the hydrogenated starch hydrolysate syrup comprises between 22% w/w and 55% w/w higher polyols, preferably between 25% w/w and 50% w/w higher polyols,
- b) cooking at a temperature between 145°C and 170°C and low vacuum, and
- c) shaping according to stamping or deposit method.

Production of the hard candies may be carried out in batch evaporators or in continuous cookers and the process time at elevated temperature generally being up to 15 minutes, preferably in the range 5 to 10 minutes.

The process used to produce hard candies from maltitol containing syrups customarily operates at a temperature between 145°C and 170°C, preferably between 150°C and 160°C. At these temperatures it is preferable to apply a low vacuum, suitably 0.6 to 0.8 bar at the end of the heating period to facilitate water removal. The shaping of the hard candies is performed according to either one of the classically applied methods, being the 'stamping' method and the 'depositing' method, respectively. In the 'stamping' method the cooked mass is cooled to 90°C or 100°C, and eventually colour, flavour and acid are mixed in. Then the mass is further cooled to 60°C or 70°C for shaping. After further cooling the candies are wrapped. In the 'depositing' method the cooked mass is cooled to 110°C or 120°C, and eventually colour, flavour and acid are mixed in. The hot mass is dosed in moulds, further cooled and demoulded afterwards. The current invention further relates to a process wherein flavour and/or colouring matter can be added during shaping.

After further cooling, the hard candies can be wrapped. The obtained sugar-free hard candies, which are transparent, non-hygroscopic, non-cariogenic can be loose packed in boxes due to their non-stickiness.

In each method, the heating phase is followed by a cooling phase and by applying more viscous mixtures (i.e. high mass viscosity) the workability is improved, the processing time of the cooling phase is shortened, and the final viscosity is reached sooner. In principle the processing time of the cooling phase is easily reduced with one quarter of the total time compared to the processing time required to cool the mass consisting of syrups based on hydrogenated isomaltulose alone. When working with a lab cooker on quantities of about 4 kg syrup, the cooling phase can easily be shortened with about 10 to 15 minutes compared to the cooling phase where hydrogenated isomaltulose syrups are applied solely. The processing time of the cooling phase of 4 kg syrup consisting of hydrogenated isomaltulose is about 40 to 60 minutes, while with the syrups of the current invention the processing time is shortened with 10 to 15 minutes.

The mass viscosity is measured directly after cooking with an oscillating shear-disc viscometer. The measurement is started at 135°C and the sample is cooled with constant rate of 2.8°C/ minute to 94°C and the measured values are recorded by means of a potentiometric line recorder.

The mass viscosity of hard candies prepared with hydrogenated isomaltulose is around 20,000 mPa.s at 94°C and hard candies prepared with hydrogenated isomerised saccharose have a mass viscosity of about 15,000 mPa.s. In comparison, the mass viscosity of C $\star$  Maltidex C16510 (which is a syrup at 70% dry substance and consisting of 70/30 isomalt/hydrogenated starch hydrolysate mixture (= 70% hydrogenated isomaltulose powder (C $\star$  Maltidex C16500) and 30% hydrogenated starch hydrolysate syrup added at commercial base (=70% dry substance) and wherein the dry substance of hydrogenated starch hydrolysate is comprising between 22% and 31% w/w higher polyols)) is about 30,000 mPa.s at 94°C. This increased mass viscosity results in improved workability and the cooling time is reduced with 10 to 15 minutes when working in a lab cooker of 4 kg scale.

Applying syrups, which are obtained by the hydrogenation of starch hydrolysates and isomaltose or 'isomerised' saccharose wherein the dry substance of starch hydrolysate contains less than 22% w/w higher oligomers, results in hard candies having a mass viscosity below 25,000 mPa.s, namely about 23,600 mPa.s.

Hard candies with a mass viscosity of at least 25,000 mPa.s, preferably higher than 27,000 give good workability and significant reduction of the processing time of the cooling phase. The time to reach the appropriate viscosity before moulding, is shortened with one quarter of the total cooling time of syrups based on hydrogenated isomaltulose alone. Furthermore, by applying the syrups of the current invention not only the mass viscosity but also the plasticity of the mass is improved during processing.

The feedstock for preparing hard candies of the present invention may comprise between 10 to 35 % by weight of water based on the weight of the mixture, most often about 30% by weight water. This feedstock is heated to an elevated temperature until a product is obtained which preferably contains less than 5% by weight water, more preferably less than 4%, more preferably less than 2% by weight or less, based upon the weight of the candy.

Furthermore the polyol-containing feedstock of the present invention also may include flavour and/or colouring matter or any other additive commonly found in hard candy products.

The candy stickiness test is performed with the Texture Analyser TA-XT2 from Stable Micro Systems. The stickiness measurement can be done by storing the candy several minutes to hours at specific conditions of humidity and temperature and the more sticky the candy the higher the adhesive force becomes. The confectionery hygroscopicity is measured in a microclimate chamber with an accurate regulation system having the possibility to create conditions of temperature from 10°C to 98°C and relative humidity from 10 to 95%. The moisture pick-up of the samples placed in the microclimate chamber stored under specific conditions of temperature and humidity is calculated from the difference in weight of the samples after a specific storage time.

Hard candies prepared with hydrogenated isomaltulose give a stickiness of 175 g (after 10 minutes cooling) at 21°C and 52% relative humidity (RH), while hard candies prepared with C★ Maltidex C16510 (which is a syrup at 70% dry substance and consisting of 70/30 isomalt/hydrogenated starch hydrolysate mixture (= 70% hydrogenated isomaltulose powder (C★ Maltidex C16500) and 30% hydrogenated starch hydrolysate syrup added at commercial base (=70% dry substance) and wherein the dry substance of hydrogenated starch hydrolysate is comprising between 22% and 31% w/w higher polyols) have under similar conditions a candy surface stickiness of 171 g.

Applying syrups, which are obtained by the hydrogenation of starch hydrolysates and isomaltose or 'isomerised' saccharose wherein the dry substance of starch hydrolysate contains less than 22% w/w higher oligomers, gives hard candies with surface stickiness (21°C at 52% RH after 10 minutes cooling) of 215 g.

Stickiness of hard candies prepared from hydrogenated isomerised saccharose is determined as being 173 g at 21°C (10 minutes cooling) and 49% RH. Under the same conditions, hard candies prepared with C★ Maltidex C16510 (which is a syrup at 70% dry substance and consisting of 70/30 isomalt/hydrogenated starch hydrolysate mixture (= 70% hydrogenated isomaltulose powder (C★ Maltidex C16500) and 30% hydrogenated starch hydrolysate syrup added at commercial base (=70% dry substance) and wherein the dry substance of hydrogenated starch hydrolysate is comprising between 22% and 31% w/w higher polyols) have a candy surface stickiness of 167g.

Surprisingly the surface stickiness of hard candies prepared with C★ Maltidex C16510 is at least as low as the candy surface stickiness of hard candies prepared with

hydrogenated isomaltulose and is even better than surface stickiness of hard candies prepared with hydrogenated isomerised saccharose. Using syrups, which are obtained by the hydrogenation of mixtures comprising starch hydrolysate with less than 22% w/w higher oligomers, results in more sticky hard candies.

Relative humidity below 50% and 22-23°C are ideal conditions to store demoulded hard candies prepared with C★ Maltidex C16510, (which is a syrup at 70% dry substance and consisting of 70/30 isomalt/hydrogenated starch hydrolysate mixture (= 70% hydrogenated isomaltulose powder (C★ Maltidex C16500) and 30% hydrogenated starch hydrolysate syrup added at commercial base (=70% dry substance) and wherein the dry substance of hydrogenated starch hydrolysate is comprising between 22% and 31% w/w higher polyols). Under these conditions the candy surface stickiness should not exceed 200 g, preferably is not higher than 180 g.

The corresponding hygroscopicity (moisture pick-up) is slightly increased from 0.48% to 1.2% (after 14 days at 70% RH and 25°C) for hard candies prepared with hydrogenated isomaltulose and hard candies prepared with C★Maltidex C16510 (which is a syrup at 70% dry substance and consisting of 70/30 isomalt/hydrogenated starch hydrolysate mixture (= 70% hydrogenated isomaltulose powder (C★ Maltidex C16500) and 30% hydrogenated starch hydrolysate syrup added at commercial base (=70% dry substance) and wherein the dry substance of hydrogenated starch hydrolysate is comprising between 22% and 31% w/w higher polyols), respectively. This increase is insignificant if one considers that moisture pick-up of candies prepared with hydrogenated starch hydrolysate syrups goes up to 11%. The hygroscopicity of hard candies, which are prepared with syrups obtained by the hydrogenation of starch hydrolysates and isomaltose or 'isomerised' saccharose wherein the dry substance of starch hydrolysate contains less than 22% w/w higher oligomers, is about 1.15%. In general a moisture pick-up less than 5% after 14 days is regarded as acceptable.

Due to the low hygroscopicity of the hard candies prepared with C★ Maltidex C16510 (which is a syrup at 70% dry substance and consisting of 70/30 isomalt/hydrogenated starch hydrolysate mixture (= 70% hydrogenated isomaltulose powder (C★ Maltidex C16500) and 30% hydrogenated starch hydrolysate syrup added at commercial base (=70% dry substance) and wherein the dry substance of hydrogenated

starch hydrolysate is comprising between 22% and 31% w/w higher polyols), the shelf life is correspondingly increased.

The telemetry technique is used to determine cariogenicity and continuously records plaque pH changes *in vivo* at the enamel-plaque interface and the plaque accumulates on the inserted glass electrode. If the plaque pH stays on the "alkaline side" of 5.7 within 30 minutes after consumption, the food can be labelled 'safe for teeth', or non-cariogenic. Hard candies prepared with C★ Maltidex C16510 have pH 6 in the telemetric method and are thus non-cariogenic.

The present invention results in non-hygroscopic, non-sticky, non-cariogenic hard candies with mass viscosity of at least 25,000 mPa.s, a candy surface stickiness below 180 g (50% RH, 22-23°C) and moisture pick-up (hygroscopicity at 70% R.H and 25°C) below 1.5%. Moreover as is well known the presence of maltitol improves the flavour release.

The present invention is illustrated by way of the following examples.

#### Example 1.

##### Cooking

4 kg C★Maltidex C16510 (which is a syrup at 70% dry substance and consisting of 70/30 isomalt/hydrogenated starch hydrolysate mixture (= 70% hydrogenated isomaltulose powder (C★ Maltidex C16500) and 30% hydrogenated starch hydrolysate syrup added at commercial base (=70% dry substance) and wherein the dry substance of hydrogenated starch hydrolysate is comprising between 22% and 31% w/w higher polyols) and the composition of dry substance is 0.7 – 1.0 % w/w sorbitol, 10 - 12% w/w maltitol, 2.4 – 4.4% w/w maltotriitol, 4.6 – 6.6 % w/w higher polyols, 41.5 – 42.3 % w/w 6-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,6 GPS), 35.3 – 36.8 % w/w 1-O- $\alpha$ -D-glucopyranosyl-D-mannitol (1,1 GPM)) was placed in the lab cooker without water addition. It was cooked under vacuum at 155°C during 5 minutes to reach an end-moisture of max. 1.6%.

### Shaping

#### "Depositing" method

The cooked mass was cooled to 110-120°C and colour, flavour and acid were mixed in. The hot mass was dosed in Teflon coated aluminium moulds and allowed cooling further, followed by demoulding. After further cooling the hard candies were wrapped.

### Characterisation

#### a) Viscosity of Hard Candies

The oscillating shear-disc viscosimeter consists of two discs, which are vertically installed and face each other in parallel.

The shear-disc, which is arranged above the oscillating sample disc, is connected with a torque-measuring device. The lower disc is connected with the drive and it oscillates at an adjustable frequency and with a constant oscillating angle. A digital temperature control is done for both discs by means of a circulation thermostat with heating capacity up to 210°C and cooling coil with a capacity up to 20°C.

A small sample of the mass was brought directly after cooking on the shear disc, both discs were tilted together, and oscillating was started. The measurement was started at 135°C and cooled with constant rate of 2.8°C/ minute to 94°C, and the measured values were recorded by means of a potentiometric line recorder.

The results are displayed in Table 1.

#### b) Confectionery Hygroscopicity

#### Microclimate Chamber

The microclimate chamber is made from a stainless steel interior, with conditioned air and diffused moisture injection micropressor controlled. The door opening gives access to the total working space in which the samples are placed. The micropressor achieves precise environmental control and the distinct advantage of rapid recovery after each door opening. By means of a fan and air guide walls, a forced but moderated air circulation ensures an even airflow over the samples.

The moisture pick-up of the samples placed in the microclimate chamber stored at 70% relative humidity and at 25°C was calculated from the difference in weight of the samples after 14 days.

The results are displayed in table 1.

### c) Candy Stickiness test

The candy stickiness test is performed with the Texture Analyser TA-XT2 from Stable Micro Systems.

A moulded candy having a diameter of 40 mm and 5 mm thickness was fitted at the lower jaw of the texture analyser base. A transparent foil loop of specific dimensions (100 mm length and 12 mm width) was fitted into the upper jaw and brought on the candy surface with a force of 40 g. The loop was then pulled away from the candy surface at high speed (10 mm/sec) and the adhesive force was measured. The stickiness measurements were done at 52% relative humidity at 21°C or at 49% relative humidity and 21°C.

The results are displayed in table 1.

Table 1

	Hard candy with C★Maltidex C16510
Mass viscosity at 94°C (mPa.s):	30,000
Hygroscopicity (70% R.H, 25°C, after 14 days)	1.2%
Stickiness (at 52% RH, 21°C)	171 g

The viscosity is shown in Figure 1.

The confectionery hygroscopicity is displayed in Figure 2.

The more sticky the candy the higher the adhesive force (here expressed in g).

The hard candies have high mass viscosity, and are non-hygroscopic and non-sticky.

### Telemetric method

The cariogenicity of hard candies prepared with C $\ddagger$ Maltidex C16510 is determined with the telemetric method.

The telemetry technique continuously records plaque pH changes *in vivo* at the enamel-plaque interface and the plaque accumulates on the inserted glass electrode. If the plaque pH stays on the "alkaline side" of 5.7 within 30 minutes after consumption, the food can be labelled 'safe for teeth', or non-cariogenic.

Figure 3 shows the telemetric evaluation of hard candies prepared with C<sub>16</sub>Maltidex C16510, followed by sucrose intake.

After consumption of hard candies prepared with C $\star$ Maltidex C16510 pH stays above 6 and the hard candies are thus non-cariogenic.

### Example 2.

Cooking

4 kg syrup (d.s. = 70% and wherein the composition of dry substance is: 0.3-0.7 % w/w sorbitol, 6.3-8.4% w/w maltitol, 1.3 – 3.4% w/w maltotriitol, 2.7 – 4.7 % w/w higher polyols, 45.2 – 46.4 % w/w 6-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,6 GPS), 38.4 – 40.1 % w/w 1-O- $\alpha$ -D-glucopyranosyl-D-mannitol (1,1 GPM)) was placed in the lab cooker without water addition. It was cooked under vacuum at 155°C during 5 minutes to reach an end-moisture of max. 1.5%.

Shaping of the hard candies was similar as in example 1. The hard candies were characterised by measuring the mass viscosity, hygroscopicity, and stickiness according to the methods described in example 1.

The obtained results are displayed in Table 2.

**Table 2**

	Hard candy with syrup of example 2
<u>Mass viscosity at 94°C (mPa.s):</u>	25,000
<u>Hygroscopicity (70% R.H, 25°C, after 14 days)</u>	0.96%
Stickiness (at 52% RH, 21°C)	176 g

The hard candies have high mass viscosity, and are non-hygroscopic and non-sticky.

#### Comparative example 1.

##### Cooking

4 kg hydrogenated isomaltulose powder (C★ Maltidex C16500) was first mixed with 1.3 kg water and pre-heated to 90-100°C. Then the mixture was cooked up in the lab cooker under vacuum at 155°C during 5 minutes to reach an end-moisture of 1.6%.

Shaping of the hard candies was similar as in example 1. The hard candies were characterised by measuring the mass viscosity, hygroscopicity, and stickiness according to the methods described in example 1.

The obtained results are displayed in Table 3.

**Table 3**

	Hard candy with C★Maltidex C16500
<u>Mass viscosity at 94°C (mPa.s):</u>	20,000
<u>Hygroscopicity (70% R.H, 25°C, after 14 days)</u>	0.48%
Stickiness (at 52% RH, 21°C)	175 g
Stickiness (at 49% RH, 21°C)	167 g

The hard candies are non-hygroscopic, non-sticky but the mass viscosity is too low.

Comparative Example 2.Cooking

4 kg hydrogenated starch hydrolysate syrup at 70% dry substance (C★ Maltidex L16303, M16311 or H16323) was placed in the lab cooker without water addition. It was cooked under vacuum at 168°C during 5 minutes to reach an end-moisture of max. 1%.

Shaping of the hard candies was similar as in example 1. The hard candies were characterised by measuring the mass viscosity, hygroscopicity, and stickiness according to the methods described in example 1.

The obtained results are displayed in Table 4.

Table 4

	Hard candy with hydrogenated starch hydrolysate syrup
Mass viscosity at 94°C (mPa.s):	533,029
Hygroscopicity (70% R.H, 25°C, after 14 days)	11%

The hard candies are very hygroscopic.

Comparative Example 3.Cooking

4 kg isomalt/hydrogenated starch hydrolysate syrup wherein the dry substance comprises below 22% w/w higher polyols (d.s. = 70% and dry substance consists of between 41.5-42.3% w/w 6-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,6 GPS), between 35.3 – 36.8% w/w 1-O- $\alpha$ -D-glucopyranosyl-D-mannitol (1,1 GPM), between 0.8 – 1.0% w/w sorbitol, between 10.5 – 12.5% w/w maltitol, between 3.3 – 5.3% w/w maltotriitol, and between 3.1 – 5.1% w/w higher polyols) was placed in the lab cooker without water addition. It was cooked under vacuum at 155°C during 5 minutes to reach an end-moisture of max. 1.6%.

Shaping of the hard candies was similar as in example 1. The hard candies were characterised by measuring the mass viscosity, hygroscopicity, and stickiness according to the methods described in example 1.

The obtained results are displayed in Table 5.

Table 5

	Hard candy with syrup of comparative example 3
Mass viscosity at 94°C (mPa.s):	23,600
Hygroscopicity (70% R.H, 25°C, after 14 days)	1.15%
Stickiness (at 52% RH, 21°C)	215 g

The hard candies are non-hygroscopic, but sticky and the mass viscosity is too low.

#### Comparative Example 4.

##### Cooking

4 kg hydrogenated isomerised saccharose syrup (d.s. = 70% and dry substance comprises 43.6% 6-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,6 GPS), 49.2% 1-O- $\alpha$ -D-glucopyranosyl-D-mannitol (1,1 GPM) and 3.1% 1-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,1 GPS)) was placed in the lab cooker without water addition. It was cooked under vacuum at 155°C during 5 minutes to reach an end-moisture of max. 1.6%.

Shaping of the hard candies was similar as in example 1. The hard candies were characterised by measuring the mass viscosity, hygroscopicity, and stickiness according to the methods described in example 1.

The obtained results are displayed in Table 6.

Table 6

	Hard candy with hydrogenated 'isomerised' saccharose
Mass viscosity at 94°C (mPa.s):	15,000
Hygroscopicity (70% R.H, 25°C, after 11 days)	0.96%
Stickiness (at 49% RH, 21°C)	173 g

The hard candies are non-hygroscopic, non-sticky, but the mass viscosity is too low.

AA.34

CLAIMS

1. A syrup (1) having a dry substance of from 60 to 80% consisting of a mixture of hydrogenated starch hydrolysate syrup (2) and isomalt powder or isomalt syrup (3) characterised in that:
  - a) the dry substance of the syrup (1) consists of from 14 to 25% w/w hydrogenated starch hydrolysate syrup (2) (dry substance) wherein the dry substance of said hydrogenated starch hydrolysate syrup (2) comprises between 22 and 55% w/w higher polyols, and
  - b) the dry substance of the syrup (1) consists of from 75 to 86% w/w isomalt, and
  - c) the dry substance of the syrup (1) consists of:  
7 to 52% w/w 6-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,6 GPS)  
24.5 to 52% w/w 1-O- $\alpha$ -D-glucopyranosyl-D-mannitol (1,1 GPM)  
0 to 52% w/w 1-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,1 GPS)  
0 to 1.3% w/w sorbitol (DP<sub>1</sub>)  
2.8 to 13.8% w/w maltitol (DP<sub>2</sub>)  
1.5 to 4.2% w/w maltotriitol (DP<sub>3</sub>)  
3.0 to 13.5% w/w higher polyols (DP<sub>n</sub>)
2. A syrup according to claim 1 characterised in that said syrup has a dry substance of 70%.
3. A sugar-free non-cariogenic hard candy comprising a syrup according to claims 1 or 2.
4. A sugar-free non-cariogenic hard candy according to claim 3 characterised in that the mass viscosity is at least 25,000 mPa.s, and that the candy surface stickiness is below 180g (50% RH, 22-23°C) and the moisture pick-up (hygroscopicity at 70% RH and 25°C) after 14 days is below 1.5%.

- A4.34
5. A process for the production of a sugar-free non-cariogenic hard candy comprising the following steps:
    - a) preparing a syrup (1) having a dry substance of from 60 to 80% consisting of a mixture of hydrogenated starch hydrolysate syrup (2) and isomalt powder or isomalt syrup (3) characterised in that the dry substance of the syrup (1) consists of from 14 to 25% w/w hydrogenated starch hydrolysate syrup (2) (dry substance) wherein the dry substance of said hydrogenated starch hydrolysate syrup (2) comprises between 22 and 55% w/w higher polyols, and the dry substance of the syrup (1) consists of from 75 to 86% w/w isomalt, and the dry substance of the syrup (1) consists of 7 to 52% w/w 6-O- $\alpha$ -D-glucopyranosyl-D-sorbitol (1,6 GPS), 24.5 to 52% w/w 1-O- $\alpha$ -D-glucopyranosyl-D-mannitol (1,1 GPM), 0 to 52% w/w 1-O- $\alpha$ -glucopyranosyl-D-sorbitol (1,1 GPS), 0 to 1.3% w/w sorbitol (DP<sub>1</sub>), 2.8 to 13.8% w/w maltitol (DP<sub>2</sub>), 1.5 to 4.2% w/w maltotriitol (DP<sub>3</sub>), 3.0 to 13.5% w/w higher polyols (DP<sub>n</sub>),
    - b) cooking at a temperature between 145 and 170°C and low vacuum, and
    - c) shaping according to stamping or deposit method.
  6. A process according to claim 5 characterised in that flavour and/or colouring matter is added during shaping.
  7. A process according to claims 5 or 6 characterised in that the syrup comprises between 10 to 35% by weight water.
  8. A process according to any one of claims 5 to 7 characterised in that the water content of the hard candy is less than 4% by weight, preferably less than 2% by weight or less, based upon the weight of the hard candy.

AMENDED SHEET

### Abstract

The present invention discloses a syrup at dry substance between 60% to 80%, consisting of a mixture of hydrogenated starch hydrolysate syrup and isomalt powder or isomalt syrup wherein the dry substance of the hydrogenated starch hydrolysate syrup comprises between 22% w/w and 55% w/w higher polyols, preferably between 25% w/w and 50% w/w higher polyols. Non-sticky, non-hygroscopic and non-cariogenic sugar-free hard candies comprising aforementioned syrup and the corresponding process are disclosed.

1/3

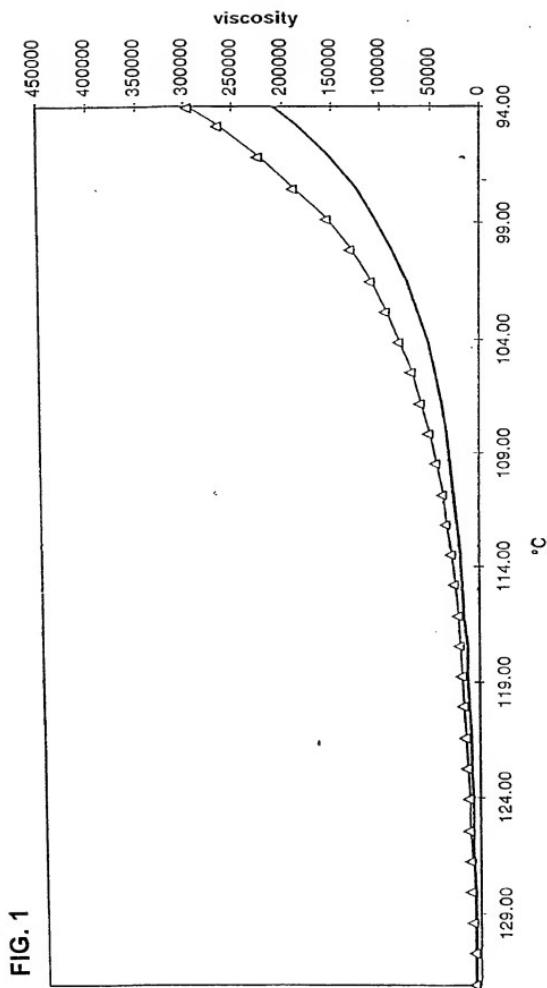


FIG. 2

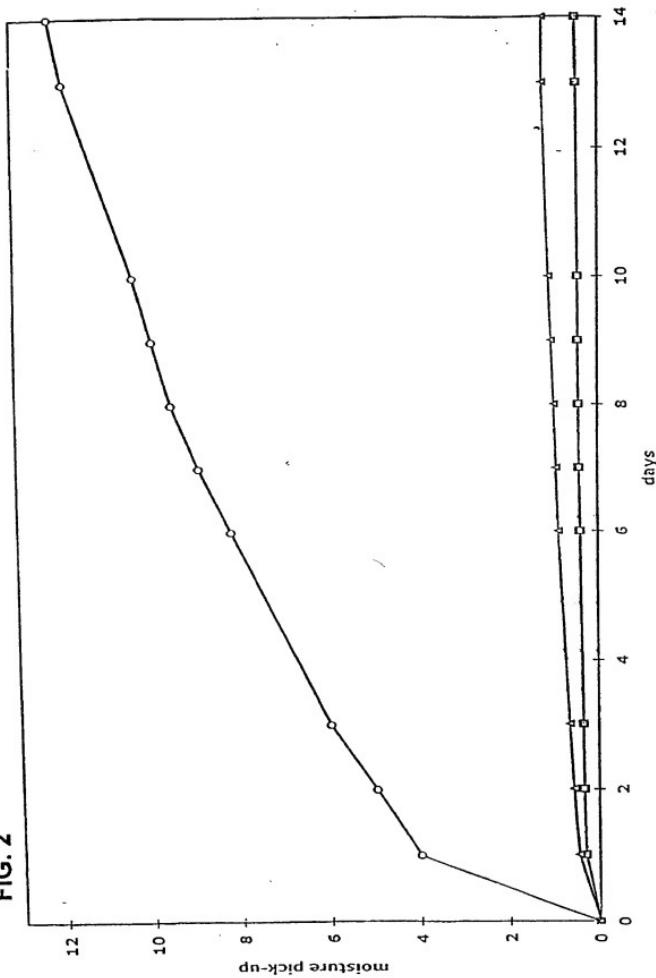
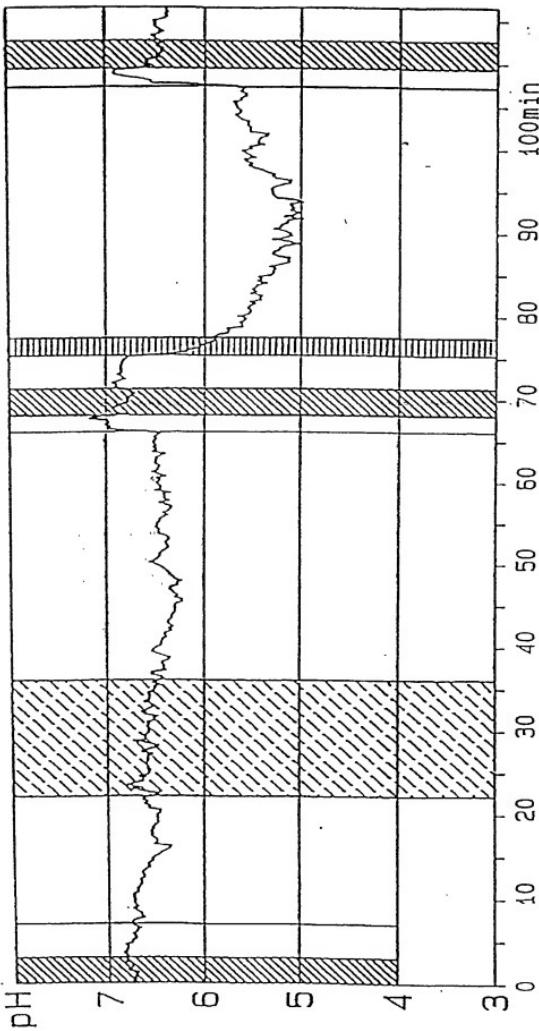


FIG. 3  
Hard Candy  
15mL  
0.3mol/L  
Sucrose



FOR UTILITY/DESIGN  
CIP/PCT NATIONAL/PLANT  
ORIGINAL/SUBSTITUTE/SUPPLEMENTAL  
DECLARATIONS

RULE 63 (37 C.F.R. 1.63)  
DECLARATION AND POWER OF ATTORNEY  
FOR PATENT APPLICATION  
IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

As a below named inventor, I hereby declare that my residence, post office address and citizenship are as stated below next to my name, and I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

**Preparation of Sugar-free Hard Candy**

(Title of Invention)

the specification of which (check applicable box(es)):

- A.  is attached hereto.  
 B.  was filed on \_\_\_\_\_ as U.S. Application No. \_\_\_\_\_  
 C.  was filed as PCT International Application Number PCT/EP00/06016 on 28/06/2000  
 and (if applicable to U.S. or PCT application) was amended on 30/4/2001

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment specifically referred to above. I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, §1.56. I hereby claim foreign priority benefits under Title 35, United States Code, §119(a)-(d) or §365(b) of any foreign application(s) for patent or inventor's certificate, or §365(a) of any PCT international application which designated at least one country other than the United States of America, listed below, and I have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or any PCT international application, on this invention filed by me or my legal representatives or assigns and having a filing date before that of the application on which priority is claimed:

PRIOR FOREIGN APPLICATION(S)

Number(s)	Country	Date/Month/Year Filed	Date First Laid-open or Published	Date Patented or Granted	Priority NOT Claimed
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GB9915883.4 08/07/1999

If more prior art exists, x box at bottom and continue on attached page.

Except as noted below, I hereby claim domestic priority benefit under 35 U.S.C. 119(e) or 120 and/or 365(c) of the indicated United States applications listed below and PCT international applications listed above or below and, if this is a continuation-in-part (CIP) application, insofar as the subject matter disclosed and claimed in this application is in addition to that disclosed in such prior applications, I acknowledge the duty to disclose all information known to me to be material to patentability as defined in 37 C.F.R. 1.56 which became available between the filing date of each such prior application and the national or PCT international filing date of this application:

PRIOR U.S. PROVISIONAL, NONPROVISIONAL AND/OR PCT APPLICATION(S)	Status
Appn. No. (series code/serial no.)	Day/Month/Year Filed pending, abandoned, patented Priority NOT Claimed

As a named inventor, I hereby appoint the practitioners associated with Customer Number 22242, with full power of substitution and revocation, to prosecute this application and to transact all business in the United States Patent and Trademark Office connected therewith, and request that all correspondence and telephone calls in respect to this application be directed to FITCH, EVEN, TABIN & FLANNERY, Suite 1600, 120 South LaSalle Street, Chicago, Illinois 60603-3406, Telephone No. (312) 577-7000, Facsimile No. (312) 577-7007, CUSTOMER NUMBER 22242.



2 2 2 4 2

I hereby declare that all statements made herein of my own knowledge are true, and that all statements made herein on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity or enforceability of the application or any patent issued thereon.

Additional inventors, see attached pages.

Additional foreign prior art on attached page (incorporated herein by reference)

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1003025-2021UE  
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City and Country for others)

Post Office Address:

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Citizenship:

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**LUDGREN, OGDEN**  
**Attorney Docket No.**

**Supplemental Data Priority Sheet**

Additional foreign application numbers:

Prior Foreign Application Number(s)	Foreign Country	Priority Filing Date	Certified Copy Attached Not Claimed	Yes	No
				<input type="checkbox"/>	<input type="checkbox"/>
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				<input type="checkbox"/>	<input type="checkbox"/>

Additional provisional application numbers:

Provisional Application Number(s)      Provisional Application Filing Date

Additional U.S. or PCT international application numbers:

Prior U.S. Application Number    Prior PCT International Application Number    U.S. or PCT International Application    Patent Number (if applicable)    Filing Date of